PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in or relating to Photographic Processes

We, INTERNATIONAL BUSINESS MACHINES CORPORATION, a Corporation organised and existing under the Laws of the State of New York, United States of America, of Armonk, New York 10504, State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention concerns improvements in or relating to photographic processes for the preparation of electrically-conductive surfaces on bases. More particularly though not exclusively it relates to the photographic preparation of so-called printed wiring upon a non-conductive base.

The term "printed wiring" is widely used to describe electrically conductive elements formed on or in the surface of a non-conductive base, and in particular is used in connection with electrically-conductive circuits wherein the conductors, and perhaps also other circuit elements such as resistors, are formed on or in the surface of a base of an insulating material, other electrical components being thereafter attached whenever necessary to complete the circuit. The use of printed wiring greatly simplifies the assembly and miniturisation of such electronic equipment.

A method for the preparation of printed wiring is as follows. An insulating base is first coated with a conductive metal such as copper, the metal coating is lacquered with a light-sensitive etch-resist, and the resist is finally exposed to light through a mask which is transparent wherever a conductor is ultimately desired but otherwise opaque. Such a mask can be prepared photographically as a preliminary step, by drawing out the desired pattern of conductors on an enlarged scale and then photographing this drawing on the scale ultimately required using a high-contrast photographic film material, which on development gives a photographic image of the drawing of the desired actual size. The resist, such as Kodak P.C. Resist (Kodak is a Registered Trade Mark) is one which has the property when unexposed to light of being soluble in certain organic solvents, such as the commercially available Kodak P.C. Resist Developer, but insoluble therein after light exposure. After the resist has been exposed through the mask it is subjected to treatment with the appropriate solvent which therefore leaves a pattern of undissolved varnish upon the surface of the metal coating corresponding to the original drawing the remainder of the resist having been dissolved away. After this solvent treatment the surface is treated with an aqueous metal etchant solution which etches away any metal left unprotected by the resist so that a pattern of metal is left on the non-conductive base forming the desired wiring elements.

This existing preparative method for printed wiring is unduly complex and time consuming since it involves several stages, and necessitates passing from a non-aqueous organic solvent to an aqueous metal etchant solution. We have now discovered a process generally applicable for the preparation of conductive surfaces on bases and of especial value in the preparation of printed wiring on non-conductive bases since it enables them to be prepared by a direct photographic technique which not only avoids the need to pass from one phase to another but also is capable of high speed automation. Moreover, the materials used in this process are commercially available and need not be made up specially.

According to the invention there is provided a process for the preparation of an electrically-conductive surface on a base, in which a radiation sensitive silver halide emulsion supported on said base is subjected to partial radiation exposure, and developed, the part of the emul-

[*Price 4s. 6d.*]

sion thus exposed and developed is removed by treatment with bleach-etch solution and the underlying residual unexposed emulsion is then fully exposed to radiation and developed so as to form a conductive layer.

At this point it may be noted that, without wishing to be limited in this respect, it is our belief that it is only the surface of the residual unexposed emulsion which becomes electrically conductive, not its entire thickness.

While the process of this invention may be applied to bases of an electrically conductive or semi-conductive material — thus for instance, connections may be made to semi-conductor chips by this process in order to prepare transistors and similar devices — we prefer in general to apply the process to bases of a substantially electrically non-conductive material, particularly in the preparation of printed wiring and for convenience the base will hereafter be referred to as a "non-conductive base".

The non-conductive bases used in the process of this invention may be any to which it is possible to apply a film of silver halide emulsion, provided only that they can withstand normal photographic processing techniques, including the bleach-etch treatment. They may thus include ceramics, mica, wood and paper of adequate wet strength. The process is preferably carried out with light-sensitive emulsions exposed to light, and therefore those already conventional in the photographic field, above all glass and especially synthetic resins. Preferred base materials are Melinex; polyvinyl chloride, cellulose acetate or triacetate and 'Perspex'. (Perspex and Melinex are Registered Trade Marks).

Any radiation sensitive silver halide emulsion can be used, though as indicated for convenience a light sensitive emulsion is preferred. Accordingly we shall hereinafter describe this invention in terms of exposure to light, but it is to be understood that where appropriate other infra-red or ultra-violet radiation can be employed. In the preparation of printed wiring it is advantageous to employ a high-contrast orthochromatic silver halide emulsion, and those conventionally utilized in photography are thus currently preferred, notably emulsions having a γ of 5 or more.

The specific preferred materials to which the process of this invention is applied are those commercially available as 'Kodalith (Registered Trade Mark) type 3 film', on an Estar (Registered Trade Mark) or glass film base.

The partial light exposure to which the silver halide emulsion must be subjected is to be understood as being one of such controlled intensity and duration that light penetrates partly but not wholly through the layer of emulsion, thus say more than 1% and less than 99% of the full thickness of the emulsion layer.

Where reference is made to a full exposure, while this in principle can include an exposure of such great duration as to convert the silver halide to silver merely by photochemical action, in practice it will normally imply only an exposure sufficient to create a latent image throughout the emulsion thickness.

Control of the exposure is most easily exercised by adjustment of the exposure time using illumination of standard intensity. In practice, using an intensity such as to give an exposure time of reasonably short duration, it will be possible conveniently to control the partial light exposure with a margin of error of approximately 10% and the practical limits for the partial exposure are then from 10% to 90% of a full exposure. As regards ease of control it is best to keep the partial exposure within the 25%—75% range, and indeed we prefer to aim at a partial exposure of 50% $\pm$ 10%. However, if ease of control can be dispensed with, it is then better to employ partial exposures approaching full exposure, since the underlying residual emulsion which separates the conductive layer from the non-conductive base is then of less thickness and consequently the adhesion between the conductive layer and the non-conductive base tends to be better than when only slight partial exposures are employed. As regards adhesion of the conductive layer to the non-conductive base, which for instance is of major importance in the production of printed wiring, it is therefore advantageous to use a partial exposure of at least 50%, preferably 60%—90%, and indeed we prefer to aim at a partial exposure of 75% $\pm$ 10%.

The development of the part of the emulsion exposed during the partial exposure may be a physical or chemical development, but it is most convenient by far to use any standard chemical photographic developing solution, for instance those sold under the Registered Trade Marks "Triolith" and "Kodak" D. 19.B.

The bleach-etch solutions used in the treatment to remove the partially exposed emulsion are in themselves well known in conventional photographic processing. Usually they contain copper sulphate and hydrogen peroxide. While the mechanism of the reaction caused by such bleach-etch solutions is not fully understood, it is well established that if an exposed and developed silver halide emulsion is subjected to treatment with bleach-etch solution then, wherever a silver image has been formed, the gelatine immediately surrounding the silver grains is loosened and can be removed by swabbing or agitation, but where there is no developed silver image no such removal takes place. The term "the bleach-etch treatment" is used herein to describe the conventional step of bleaching and etching away fully exposed and developed silver halide emulsion, using swabbing or other abrasive means if necessary to aid in complete removal of the silver image and surrounding gelatine. It is however to be

borne in mind that the emulsion interface surface, prepared by the novel bleach-etching treatment of a partially-exposed and developed emulsion which forms a fundamental feature of this invention, can apparently be easily damaged by rough handling, resulting in an inability to produce the desired conductive surface, and thus the term "treatment with bleach etch solution" is used hereinafter wherever the emulsion interface surface must not be damaged and therefore swabbing or like abrasive treatments must at most be very gentle and preferably should be avoided altogether, though some measure of agitation or induced turbulence may be permissible.

After full exposure, it is necessary to subject the product to conventional photographic development. Again development may be either physical or chemical, but the latter is preferred, and it is indeed advantageous at this stage to employ an energetic developer such as those commercially available as "Kodak" D.G.10 or D.158. Subsequent hardening of the emulsion is often advantageous, though this is not a necessary step.

It is surprising that the process of this invention does yield an electrically conductive surface in view of the recognised fact that normal processing of photographic film material certainly does not at any stage yield an electrically conductive layer, despite the formation of metallic silver in the image.

As thus far described the process of this invention is broadly useful for any purpose where it is desired to form a conductive surface layer on a non-conductive, semi-conductive or conductive base. Such a process using a non-conductive base might be required in the manufacture of paper capacitor rolls or even merely in the metallization of say ceramic surfaces for decorative effect. At this point it may be noted that, although the conductive surface layer thus formed is silver, the process is not limited only to the formation of layers of this metal since once a rudimentary silver layer has been created it can readily in a further step or steps be overlaid with other chemically or electrolytically deposited metals. While this is feasible when the conductive surface is formed on a conductive base, it is especially preferred that this further step should be applied to semi-conductive or non-conductive base materials and this will indeed be described hereinafter.

The process of the invention is indeed primarily of value in the production of printed wiring on a non-conductive base. For that purpose it will be appreciated that, starting with a light-sensitive silver halide emulsion coated evenly upon a base, it is desired to form a conductive layer only in certain lines or areas which we will call "ultimately conductive areas", and not in others which we will call "ultimately non-conductive areas". It is therefore important to note that, provided that in carrying out the process it is only the ultimately conductive areas which are subjected to the partial light exposure, it is possible whether the ultimately non-conductive areas are either fully exposed or left unexposed to achieve the desired ultimate result, although the procedures employed will naturally differ in detail and the results attained will vary somewhat in certain inessential respects.

All the various possible procedures involve a discriminating exposure, that is to say a partial light exposure of the ultimately conductive areas, and either a full exposure or non-exposure of the ultimaely non-conductive areas. It should be noted that the term "full exposure" embraces successive partial exposures sufficient to expose the silver halide emulsion throughout its entire thickness. It should also be noted that where an exposure is followed by the bleach etch treatment, it is to be inferred that the exposed film is developed first, and then bleach etched. All partial or full exposures may be performed either by focussing a suitable projected image of chosen intensity for the appropriate time upon the silver halide emulsion or by exposing the emulsion to light of chosen intensity for the appropriate time through a superimposed mask. The latter technique is usually preferable in mass production, and moreover lends itself most readily to simple explanation. For these reasons the process of the invention will hereinafter be described in terms of the use of a mask but it is to be borne in mind that where reference is subsequently made to a mask this, if the context allows, is to be regarded as also embracing a projected image.

The mask, prepared by a photographic procedure similar to that currently employed, may be either negative or positive in character. By a negative mask we mean one in which the mask areas corresponding to the ultimately non-conductive areas of the printed wiring are relatively or absolutely transparent and the mask areas corresponding to the ultimately conductive areas of the printed wiring are relatively or absolutely opaque. By a positive mask we mean one in which the mask areas corresponding to the ultimately non-conductive areas of the printed wiring are absolutely opaque, and the mask areas corresponding to the ultimately conductive areas of the printed wiring are relatively or absolutely transparent.

Using a positive mask, the simplest of the available procedures involves carrying out a single discriminating exposure through the positive mask for such a period at the chosen intensity as to effect a partial exposure of the silver halide emulsion in the ultimately conductive areas, while leaving unexposed the ultimately non-conductive areas which are hidden from exposure behind the opaque areas of the mask. The treatment with bleach etch solution thus leaves the ultimately non-conductive areas unaffected, but in the ultimately conductive

areas removes the exposed part of the gelatine/silver emulsion so that the underlying unexposed residual emulsion with its surface conditioned for conduction is somewhat recessed into channels in the non-conductive gelatine emulsion matrix.

The main disadvantage of this procedure lies in the fact that, if subsequently both the ultimately conductive and the ultimately non-conductive areas are subjected simply to an indiscriminate full exposure and development, the pattern of the rudimentary printed wiring is at best hardly discernible at this stage. Usually this is not a serious disadvantage, since the pattern will later become apparent if the rudimentary printed wiring is built up by chemical and/or electrolytic deposition of metal upon the conductive surfaces, which frequenty is desirable in subsequent manufacturing operation as will be described hereinafter.

However, if this disadvantage is to be avoided, this can be achieved by somewhat elaborating the technique. Instead of subjecting the emulsion to an indiscriminate overall exposure after the treatment with bleach etch solution, it is instead discriminatingly re-exposed through the same positive mask until the ultimately conductive areas are fully exposed, and then developed and fixed. Only the ultimately conductive areas are affected by this discriminating exposure, and therefore the ultimately non-conductive areas, being wholly unexposed, are not in any way developed and consequently, when subjected to an ordinary fixing treatment, they are freed from silver halide though not stripped of their gelatine coating. The previously formed recessed conductive areas which after development are dark in colour thus remain discernible in this light-coloured gelatine coating.

Using a negative mask the available procedures include an indiscriminate partial exposure of the whole area of the emulsion, followed by a discriminating exposure through a negative mask, preferably of the fully transparent/fully opaque type, for such a period as to complete the exposure of the ultimately non-conductive areas while leaving the ultimately conductive areas only partially exposed. Alternatively this order can be reversed, so that the discriminating exposure through the mask precedes an indiscriminate exposure of such duration as to complete the exposure of the previously partly exposed ultimately non-conductive areas while only partially exposing the ultimately conductive areas. Both these alternative procedures however involve successive exposures, one discriminating and the other indiscriminate.

To avoid the need for successive exposures when using a negative mask the procedure can be varied so as to carry out a single discriminating exposure using a film of a low gamma value through the negative mask of the transparent/semi-opaque type for such a period as completely to expose the ultimately non-conductive areas yet only partially to expose the ultimately conductive areas of the printed wiring.

All of these variants, when followed by development and the bleach-etch treatment, result in the whole thickness of gelatine/silver halide emulsion being stripped from the base in the ultimately non-conductive areas, to which therefore no further attention need be paid, while in the ultimately conductive areas only the exposed part of the gelatine/silver halide emulsion is removed by treatment with bleach-etch solution, the underlying unexposed residual emulsion remaining upstanding upon the base wth its surface conditioned for conduction.

As a minor variant of these procedures, a discriminating exposure through a negative mask with fully opaque areas can be immediately followed by the bleach-etch treatment to strip the ultimately non-conductive areas without affecting the ultimately conductive areas, and this then is followed by a further exposure, which can be indiscriminate but must be only partial, after which a further treatment with bleach-etch solution yields the desired result. In this variant it must however be borne in mind that the further exposure, being partial, must be carried out with reasonable uniformity if it is to be correctly controlled, and this uniformity of exposure is difficult to preserve if carried out upon film material wet from the preceding bleach-etch treatment since this tends to drain and dry unevenly. Care should therefore be taken either to complete drying of the film material before the further partial exposure or, better still, to carry out the further partial exposure with the film material completely submerged in water.

Whichever of these varied procedures is adopted, the final product is in essence a printed circuit, since the conductive areas secured after the treatment with bleach-etch solution followed by full exposure and development will normally carry an electric current, provided of course they are arranged in a suitable continuous pattern so as to constitute a circuit. The printed wiring conductors thus created are, however, rudimentary and for most practical purposes require to be further built up, and in some cases they can be so rudimentary that they cannot be relied on to carry an electric current till they have been further built up.

Accordingly the process of the invention will advantageously include the subsequent step or steps of growing the conductors by building up metal upon the rudimentary conductive areas, either by chemical or electrolytic deposition. In principles these deposition techniques can be used as alternatives, except where the conductive areas are so rudimentary that no electric current will flow and only chemical deposition can be used, but because anyway

printed wiring can include disconnected conductive areas (usually termed 'lands') which are electrically isolated from the remainder of the wiring until the other components of the circuit are attached, it is in fact often preferable to grow the conductors by subjecting the conductive areas, at least initially, to the electroless deposition of metal, desirably copper, using conventional chemical metal plating solutions. However in some cases the conductive areas can alternatively be subjected to electrolytic deposition, again preferably of copper, from a conventional plating electrolyte, or more frequently it may be desirable to subject the chemically metallized conductors to a final electrolytic deposition.

It will be appreciated that the growth of the conductors by metallization in this manner can result in some loss of the definition of the printed circuit. The loss is usually relatively small and in most cases negligible; but where for reasons of miniaturisation the usual loss of definition is unacceptable it can be somewhat diminished by the use of a positive rather than a negative mask during the preparation of the rudimentary printed circuit, since the recessed channels in the gelatine matrix within which the conductive areas then lie restrict the build-up of deposited metal to the upper surface of the conductive layer. Even when it is preferred to have the conductive areas upstanding upon the base rather than recessed into a gelatine matrix, this advantage of minimized loss of definition can be retained by a variant of the overall process in which the partial exposure development and subsequent treatment with bleach-etch solution, preceding full exposure and development of the ultimately conductive areas, is followed by metallization of the conductors, the full exposure and subsequent beeach-etch treatment of the ultimately non-conductive areas being deferred until after metallization of the conductive areas has been completed.

After metallization of the conductors the printed wiring is in a condition for use. It may however be subjected to further operations if desired, such as baking to eliminate any residues of gelatine, or lacquering to render the surface non-hygroscopic.

The invention of course includes articles bearing an electrically conductive surface on a base, especially printed wiring on a non-conductive base, whenever prepared in or by a process as herein described.

In order that the invention may be well understood one preferred mode of performing the process will now be described, though only by way of illustration, in the following detailed examples:—

EXAMPLE:

Stage 1 A negative mask is prepared by conventional photographic techniques, showing the desired pattern of printed wiring. Kodalith Type 3 Lithofilm on an Estar base is exposed through that mask, for a period of twenty seconds using a 100 watt 12 volt point source tungsten filament vacuum lamp, run at 14 volts.

Stage 2 The film thus exposed is developed in a photographic tray under a safelight for two minutes in Triolith (Regitered Trade Mark) developer at a temperature of 68°F, with constant agitation. The Triolith developer used is made up of one part stock solution A, one part of stock solution B and four parts of distilled water.

Stage 3 The developed film is then rinsed in running clean water for ten seconds.

Stage 4 The rinsed film is then subjected to the bleach-etch treatment by immersing it for a period of 75 seconds at a temperature of 68°F. in a bleach-etch solution made up by mixing together one part of stock solution C and one part of stock solution D. The composition of stock solutions C and D is as follows:—

Stock solution C

Cupric Sulphate Crystals	187 grms
Citric Acid Crystals	49.1 grms
Potassium Bromide Crystals and water to make one litre	16.8 grms

Stock Solution D

100 volume Hydrogen Peroxide	1 part
Water	1—7 parts

During treatment the film surface is preferably wiped carefully with cotton wool to produce a clean image.

Stage 5 The bleach-etched film is then subjected to partial re-exposure but to ensure uniformity of exposure it is first submerged in distilled water contained in a photographic tray lined with black paper. Once the water has settled to a still state, partial re-exposure is effected by turning on the room lights for a period of six seconds.

Stage 6 The partially exposed film is then developed by immersing it for 45 seconds with constant agitation in a photographic tray containing Kodak DG 10 developer at a temperature of 68°F.

Stage 7 The developed film is then rinsed for ten seconds in clean running water.

Stage 8 The rinsed film is treated with bleach-etch solution having a composition similar to that stated in stage 4 for a period of 75 seconds at a temperature of 68°F. During the treatment the solution may be agitated, but wiping with cotton wool is avoided. At this stage the use of a safelight can be dispensed with.

Stage 9 The film is washed for 60 seconds in running water, care being taken to remove all the exposed emulsion, but despite the tendency for gelatine to remain on the surface of the emulsion this is removed only by the action of the water, and no wiping of the surface with cotton wool is permissible.

Stage 10 Owing to the room lights having been turned on in stage 8 or 9, the residual emulsion has been fully exposed, and the exposed film is subjected to development for a period of two minutes in a photographic tray containing Kodak DG 10 developer at a temperature of 68°F.

Stage 11 The rudimentary printed wiring is now washed in running water for at least 10 minutes and allowed to dry under normal room temperature and humidity conditions.

Stage 12 The printed wiring is with care polished by gentle rubbing with cotton wool until all loose deposits have been removed and the surface of the wiring has acquired a metallic silver sheen.

Stage 13 The polished printed wiring is built up by electroless deposition of copper thereon using Cuposit (registered Trade Mark) coppermix 328. This material, commercially available from the Shipley Co. Inc., is believed to contain a formaldehyde reduction product of copper sulphate in solution with an alkali.

Stage 14 After electroless plating to the required extent, the printed wiring is washed until the blue colour acquired during electroless deposition completely disappears, usually after approximately ten minutes. The washed printed wiring is finally dried under normal room temperature and humidity conditions.

WHAT WE CLAIM IS:—

1. A process for the preparation of an electrically-conductive surface on a base, in which a radiation-sensitive silver halide emulsion supported on said base is subjected to partial radiation exposure and developed, the part of the emulsion thus exposed and developed is removed by treatment with bleach-etch solution, and the underlying residual unexposed emulsion is then fully exposed to radiation and developed so as to form a conductive layer.

2. A process as claimed in claim 1 in which the base is electrically non-conductive.

3. A process as claimed in claim 1 or claim 2 in which the emulsion is a light-sensitive silver halide emulsion.

4. A process as claimed in any of the preceding claims in which the base used is glass or a terephthalate synthetic resin.

5. A process as claimed in any of the preceding claims in which the emulsion employed is a high-contrast orthochromatic silver halide emulsion having a γ of at least 5.

6. A process as claimed in claim 5, in which the material to which the process is applied is the film emulsion on a glass film base.

7. A process as claimed in any of the preceding claims in which the partial light exposure to which the emulsion is subjected is of such controlled intensity and duration that light penetrates through the layer of emulsion to an extent of from 10% to 90% of a full exposure.

8. A process as claimed in claim 7, in which the partial exposure employed penetrates to an extent of 25%—75%.

9. A process as claimed in claim 8, in which for ease of control the partial exposure employed penetrates to an extent of 50% $\pm$ 10%.

10. A process as claimed in any of claims 1 to 7 in which to enhance adhesion of the conductive layer to the base the partial exposure employed penetrates to an extent of 60%—90%.

11. A process as claimed in claim 10 in which the partial exposure penetrates to an extent of 75% $\pm$ 10%.

12. A process as claimed in any of claims 1 to 11 including after full exposure the additional step of hardening the emulsion.

13. A process as claimed in any of the preceding claims for the preparation of printed wiring, in which the silver halide emulsion is subjected to discriminating exposure through a mask such as to effect partial exposure of the ultimately conductive areas and either full exposure or non-exposure of the ultimately non-conductive areas.

14. A process as claimed in claim 13, in which a single discriminating exposure is carried out through a positive mask for such a period at the chosen intensity as to effect a partial exposure of the silver halide emulsion in the ultimately conductive areas, while leaving unexposed the ultimately non-conductive areas.

15. A process as claimed in claim 14 in which after the treatment with bleach-etch solution the emulsion is discriminatingly re-exposed through a positive mask until the ultimately conductive areas are fully exposed, and then developed.

16. A process as claimed in claim 13, in which an indiscriminate partial exposure of the whole area of the emulsion is effected followed by a discriminating exposure through a negative mask or *vice versa*.

17. A process as claimed in claim 16 in which the mask is of the fully transparent/fully opaque type.

18. A process as claimed in claim 13 in which a single discriminating exposure of the emulsion is effected through a negative mask of the transparent/semi-opaque type for such

a period as completely to expose the ultimately non-conductive areas yet only partially to expose the ultimately conductive areas of the printed circuit.

19. A process as claimed in claim 13 in which a discriminating full exposure through a negative mask with fully opaque areas is followed by development and then by bleach-etch treatment, this is then followed by a partial exposure, indiscriminate or otherwise, and subsequent development, after which the treatment with bleach-etch solution yields the desired results.

20. A process as claimed in claim 19 in which the partial exposure is carried out with the film material completely submerged in water.

21. A process as claimed in any of the preceding claims, which includes the subsequent step or steps of growing the conductors by building up metal upon the rudimentary conductive areas by either chemical and/or electrolytic deposition.

22. A process as claimed in claim 21 in which the conductive areas are subjected to the electroless deposition of metal and thereafter the chemically metallised conductors are subjected to electrolytic deposition.

23. A process as claimed in claim 22 in which the electrolessly deposited metal is copper.

24. A process as claimed in claim 22 or 23 in which the electrolytically deposited metal is copper.

25. A process as claimed in claim 13 using a positive mask in which partial exposure, development, subsequent treatment with bleach-etch solution, and full exposure and development of the ultimately conductive areas is followed by metallization of the conductors, full exposure, development and subsequent bleach-etch treatment of the ultimately non-conductive areas being deferred until after metallization of the conductive areas has been completed.

26. A process as claimed in any of the preceding claims in which the conductive layer is baked to eliminate any residues of gelatine and/or lacquered to render the surface non-hygroscopic.

27. A process as claimed in any of the preceding claims and substantially as described herein, particularly with reference to the Example.

28. Articles bearing an electrically conductive surface on a non-conductive base whenever prepared in or by a process as herein described and claimed.

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